NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.281-2020

National food safety standard - Determination of Cinnamaldehyde Residues in Foods

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1 Scope

This Standard specifies the gas chromatography-mass spectrometry method for the determination of cinnamaldehyde residues in foods.

This Standard is applicable to the determination of cinnamaldehyde residues in fruits and poultry meat.

2 Principle

The cinnamaldehyde in the specimen is homogeneously extracted by ethyl acetate.

According to different sample matrices, the extracting solution is purified by primary secondary amine (PSA) solid phase adsorbent, graphitized carbon (GCB) solid phase adsorbent or eighteen carbon (C18) solid phase adsorbent; detected and confirmed by gas chromatography-mass spectrometry, and quantified by external standard method.

3 Reagents and Materials

Unless otherwise is specified, the reagents that are used in this Method indicate chromatographically pure; and the water is the Class-I water specified in GB/T 6682.

3.2 Preparation of solution

source (EI).

5.1 Preparation of the specimen

Take about 500g of the edible part of a representative sample; mash it evenly with a

grinder; put it into a clean container; seal it; then mark it. The specimen is stored at -18°C.

5.2.1 Fruit: Take 5g (accurate to 0.01g) of the specimen in a 50mL centrifuge tube; add

10mL of ethyl acetate; and extract homogeneously at 10000r/min for 1min. The extracting solution is centrifuged at 8000r/min for 5min; and transfer the upper layer ethyl acetate extracting solution; and make constant volume to 10mL for purification.

5.2.2 Poultry meat: Take 5g (accurate to 0.01g) of the specimen in a 50mL centrifuge

tube; add 10mL of water; and vortex to mix. Add 2mL of potassium ferricyanide solution and 2mL of zinc acetate solution to the extracting solution, respectively; vortex and mix well; then add 20mL of ethyl acetate; and extract homogeneously at 10000 r/min for 1min. The extracting solution is centrifuged at 8000r/min for 5min; transfer the upper layer ethyl acetate extracting solution; and make the constant volume to 20mL for purification.

5.3.1 Fruit: Respectively take 50mg of primary secondary amine (PSA) solid phase

adsorbent, 10mg of graphitized carbon black (GCB) solid phase adsorbent and 10uL toluene into a 2mL centrifuge tube with a stopper; and add 1mL of extracting solution;

vortex for 1min; stand for 5 min; pass the purified liquid through the syringe filter membrane for gas chromatography-mass spectrometry determination and confirmation.

5.3.2 Poultry meat: Respectively take 50mg of primary secondary amine (PSA) solid

phase adsorbent, 10mg of graphitized carbon black (GCB) solid phase adsorbent, 100mg of eighteen carbon (C18) solid phase adsorbent and 10uL of toluene into a 2mL specified in 5.4.1. If the sample solution and the standard solution have peaks at the same retention time, the mass spectrometry shall be confirmed. In the sample spectrum after subtracting the background, all the selected ions appear, and the ion abundance ratio of the selected ions is consistent with the relative abundance of the related ions of the standard product; and the fluctuation range is within the maximum allowable deviation of Table 2, which can determine the presence of cinnamaldehyde in the sample. The confirmed samples may be judged to be positive for cinnamaldehyde. Refer to Figure A.1 in Appendix A for the mass spectrum of cinnamaldehyde standard solution.

Table 2 -- The Maximum Allowable Deviation of the Relative Ion Abundance of

the Mass Spectrum

5.4.3 Quantitative determination

According to the content of cinnamaldehyde in the sample solution, select a standard working solution with a concentration similar to that of the cinnamaldehyde in the sample solution. The response value of cinnamaldehyde in standard working solution and sample solution shall be within the linear range determined by the instrument.

Standard working solution and sample solution are injected with equal volume respectively for determination. Under the above chromatographic conditions, the retention time of cinnamaldehyde is about 8.8min, and the total ion current chromatogram of the standard product is shown in Figure A.2. in Appendix A.

5.5 Blank test

Except that no sample is added, all shall be carried out in accordance with the above-mentioned measurement steps.

6 Calculation and Expression of Results

The content of cinnamaldehyde in the sample is calculated according to Formula (1):

Where:

X - The content of cinnamaldehyde in the sample, in mg/kg;

c - the mass concentration of the test substance in the specimen solution derived from the standard curve, in ug/mL;

Relative ion abundance (% basic peak)

Maximum allowable deviation to to